Raytheon
Integrated Defense Systems

Optical and Mechanical Properties of Nano-Composite Optical Ceramics

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Submicron and Nanostructured Ceramics

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Introduction

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- Background
  - Current MWIR transparent materials
  - Nano-composite oxides
- Processing/Microstructures
- Optical Properties
- Mechanical Properties
- Summary

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Maximizing the Optical and Mechanical Performance

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The goal is to achieve all of the following simultaneously:

<u>DARPA Goals:</u>

- **High Strength - equivalent to Sapphire**
- **Scaleable Method - able to produce 3" domes**
- **MWIR Transparent - equivalent to Spinel**

<u>Raytheon "Stretch Goal"</u>

- **MWIR Transparency - equivalent to Yttria**

To achieve these goals:

- **No pore phase (large $\Delta n \sim 0.8 =$ scatter)**
- **Minimize grain size / grain growth (G.S. $\leq \lambda/20$ for transparency)**
- **Uniform 2-phase microstructure (small $\Delta n < 0.2$)**
- **Avoid MWIR absorptions due to Si-O and Al-O bonds**

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Project Approaches

Raytheon Group
Densify nano-powders to make domes

Rutgers / UC-Davis Group
Plasma sprayed powders
Spark plasma sintering

UConn Group
Direct plasma deposition of dome shapes

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- Material test and characterization
- Optical material modeling

Background

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Sapphire (single crystal Al_2O_3) is the current MWIR dome material of choice.

- High strength
- Excellent erosion durability (rain/sand)
- High thermal shock resistance
- Low optical scatter
- Intrinsic birefringence
- Lacks full 3-5µm transparency (absorption at 5µm)
- Significant MWIR emission at elevated/operating temperatures
- High temperature mechanical properties degradation
- High cost due to single crystal growth and optical finishing

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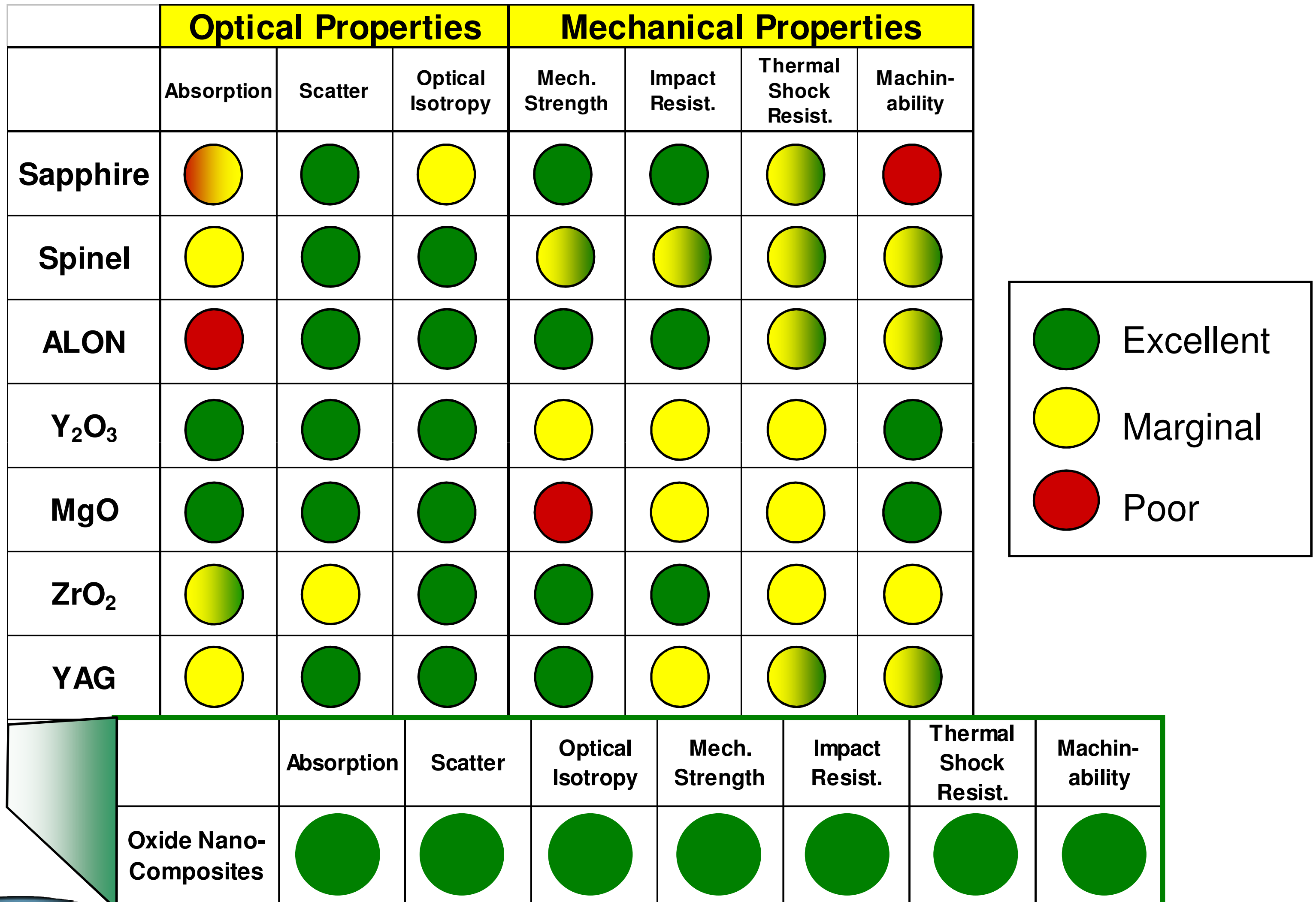
Objective:

Make much stronger dome materials and retain full MWIR transmittance



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Maximizing the Optical Performance

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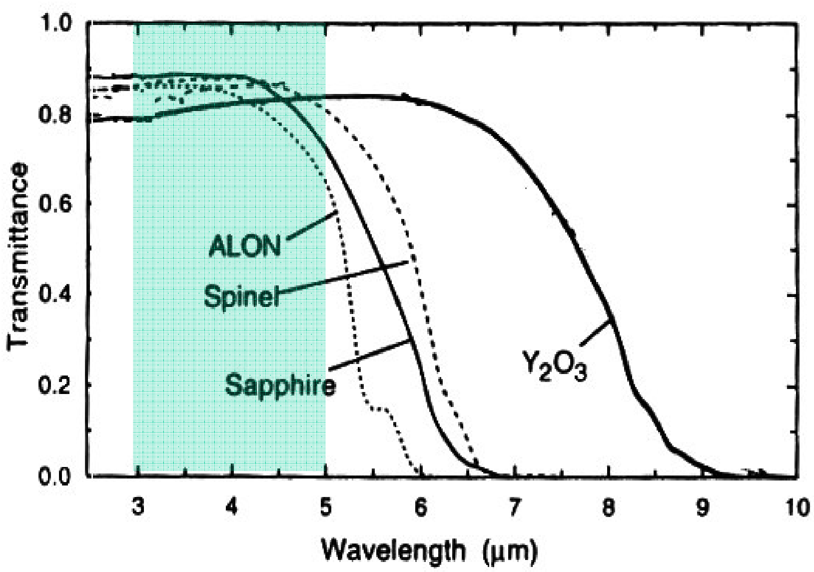
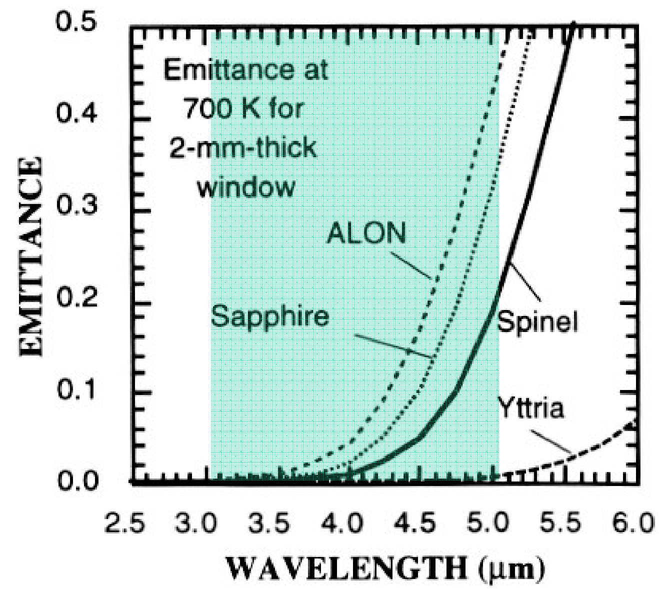
Problem: Most durable MWIR dome materials contain Al-O bonds. However, Al-O bonds absorb at $\lambda > 4$ microns

Solution: Select nanocomposite systems without Al-O bonds (Y_2O_3, MgO, ZrO_2)





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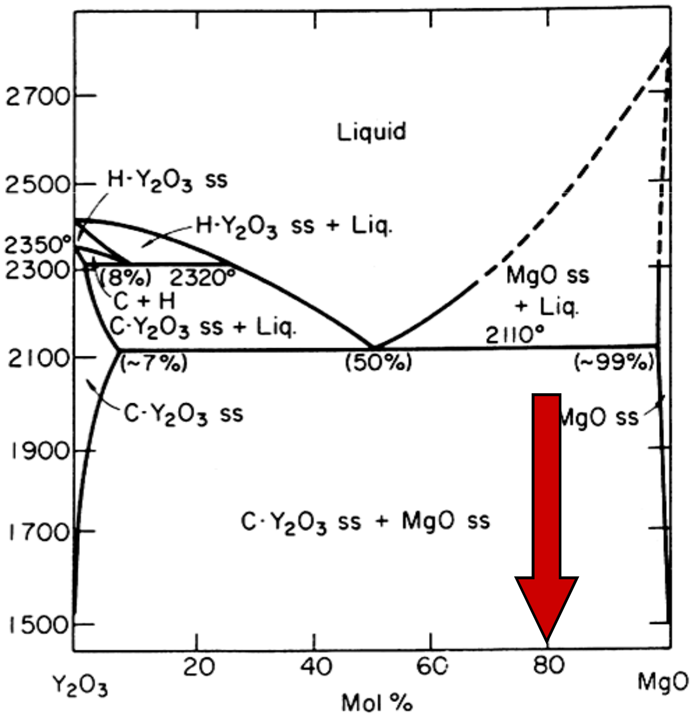
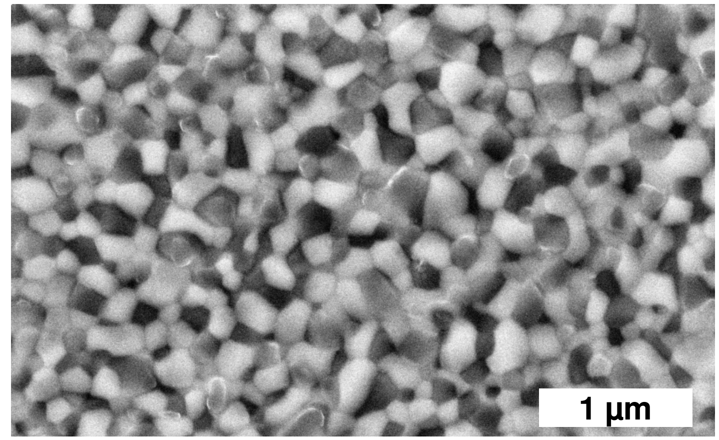
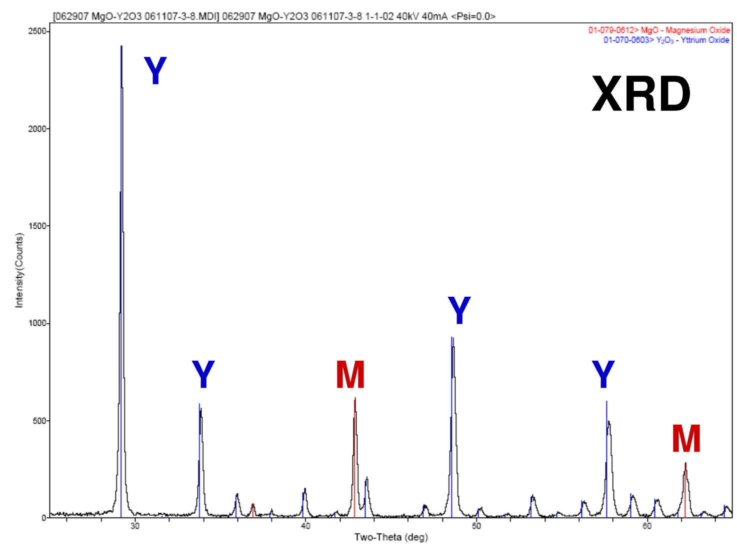
Nanocomposite Composition

Baseline Material System:
Yttria : Magnesia

Y_2O_3:MgO 20:80 Mol% → **Y_2O_3:MgO 50:50 Vol%**







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Nanocomposite Optical Ceramics:

A new class of MWIR dome materials

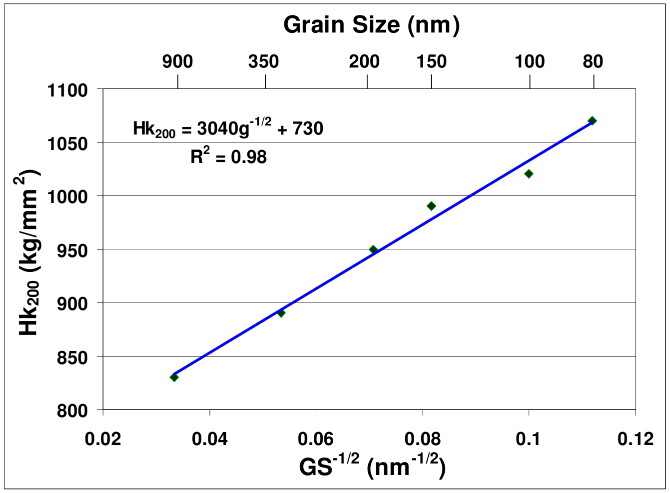
Approach: ***Reduce grain size*** **of transparent polycrystalline ceramics to *increase strength*: Hall Petch relation: $\sigma \propto (g.s.)^{-1/2}$**

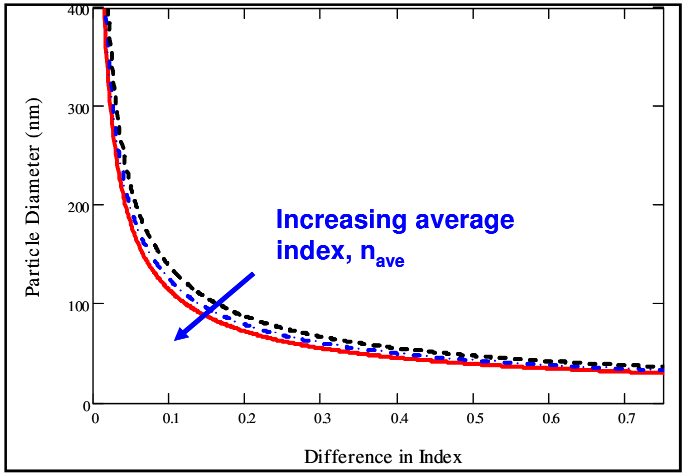
Problem: Processing conditions (high T & P) required to densify to optical clarity promote grain growth

Solution: Use significant volume fractions of two or more mutually insoluble transparent ceramics (e.g. MgO + Y_2O_3)

Problem: Refractive index differences between phases cause scattering by the grains

Solution: Reducing the grain size to < λ/20 eliminates scatter and transparency is restored: 4µm/20 = 200 nanometers !





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Nanopowder Production via Liquid Flame Spray Pyrolysis

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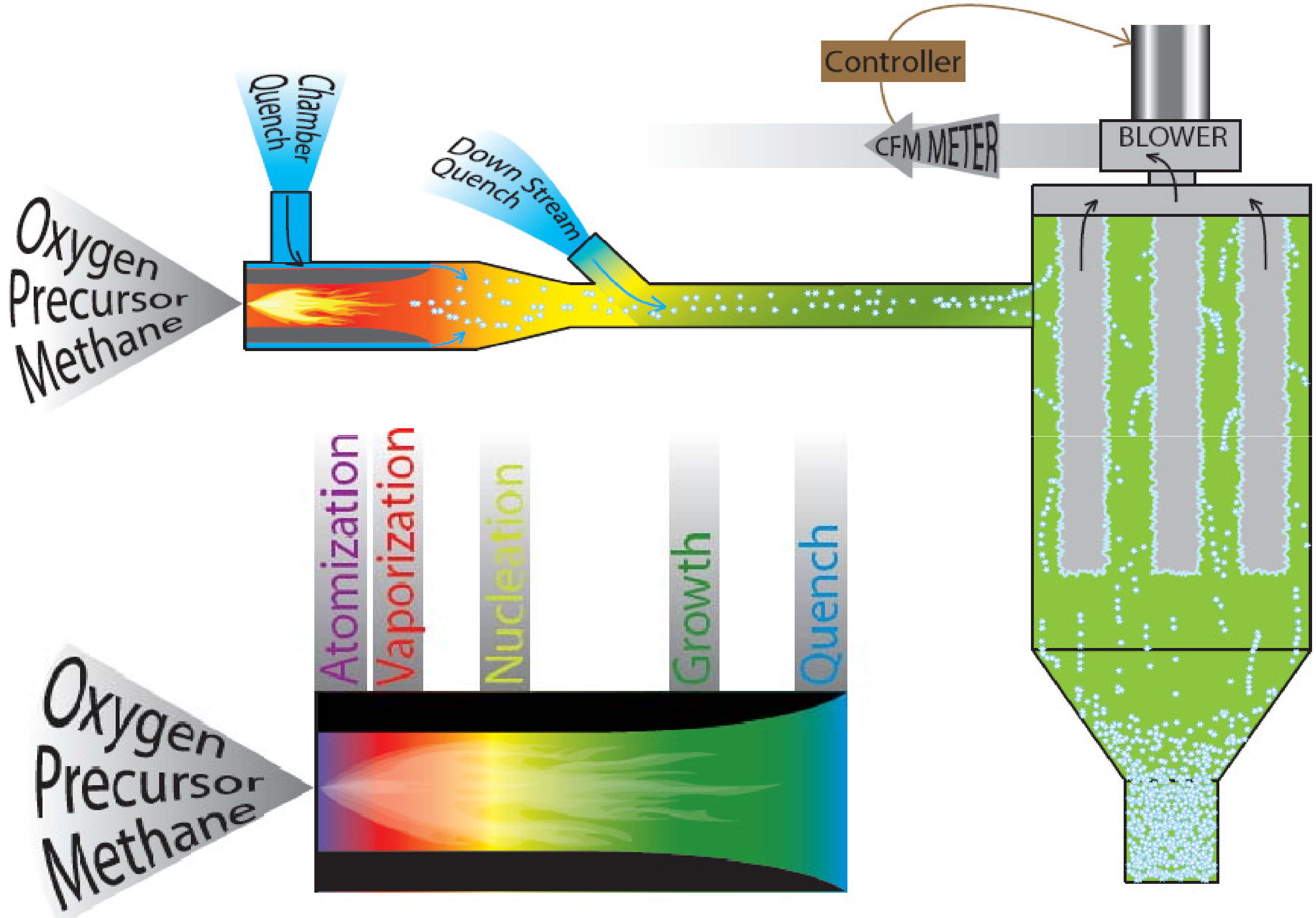

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Process Overview

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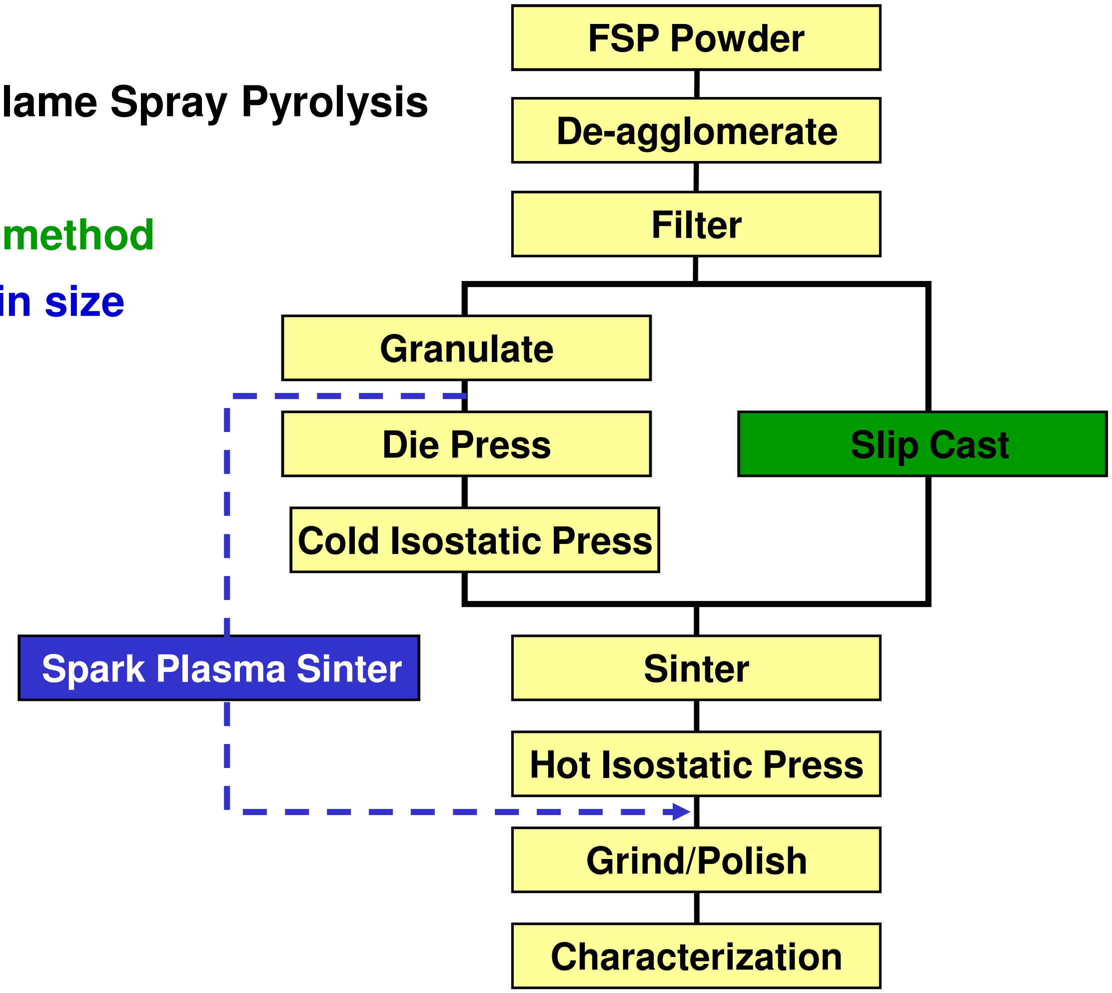
Powder Process

- Starting powders produced by Flame Spray Pyrolysis
- Densified by Sinter + HIP
- Slip Casting – alternate forming method
- SPS densification – smaller grain size



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Nanocomposite Microstructure

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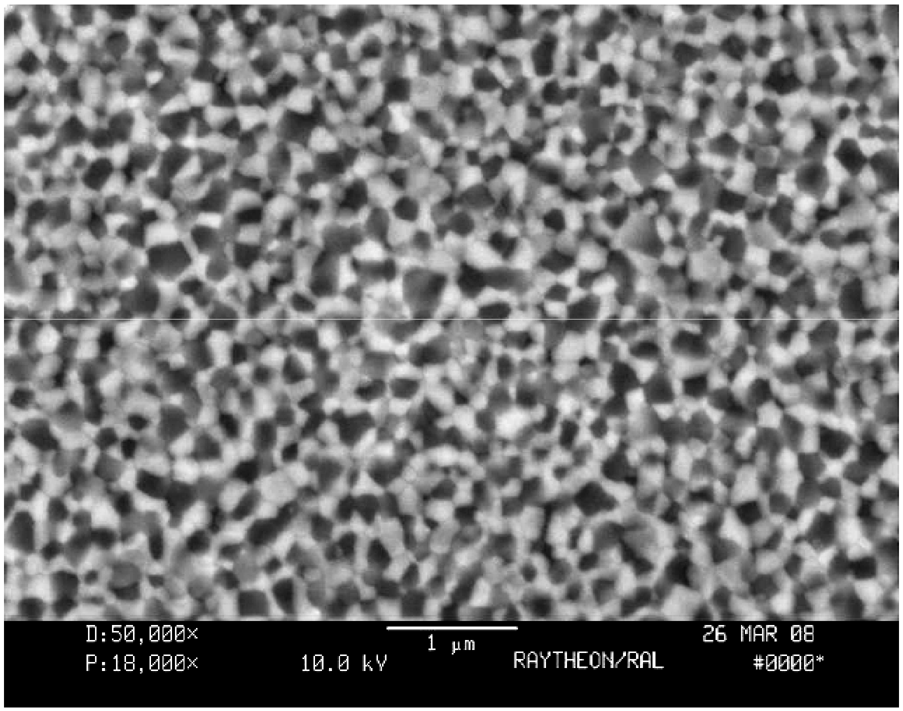
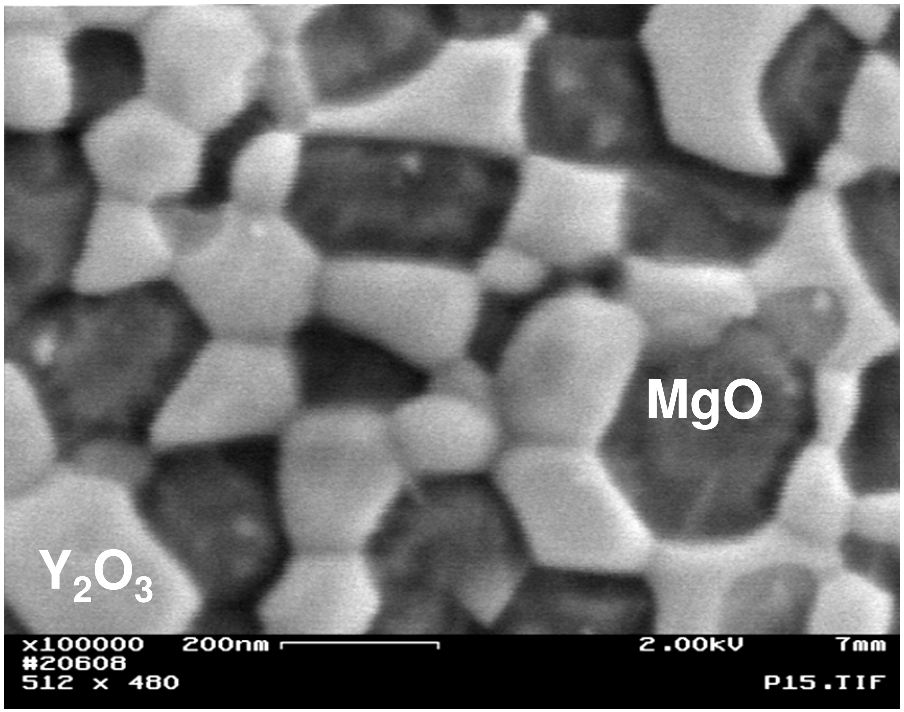
Backscattered electron images.
50:50 Vol% Yttria:Magnesia





Uniform microstructure with ~150nm grain size.

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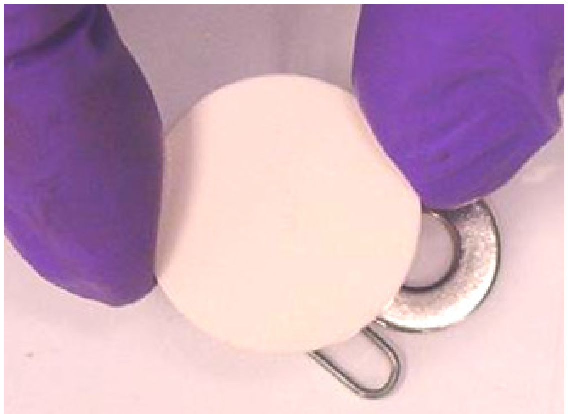
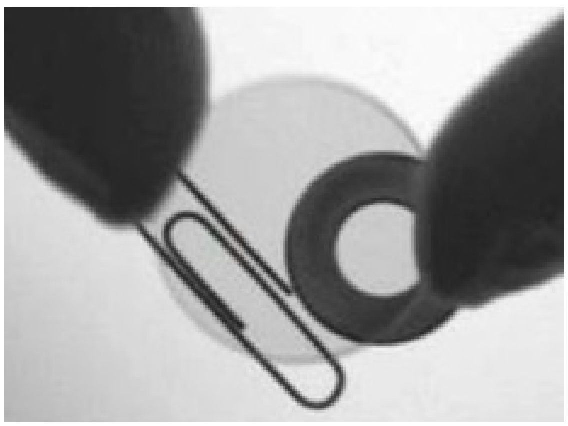
Optical Properties

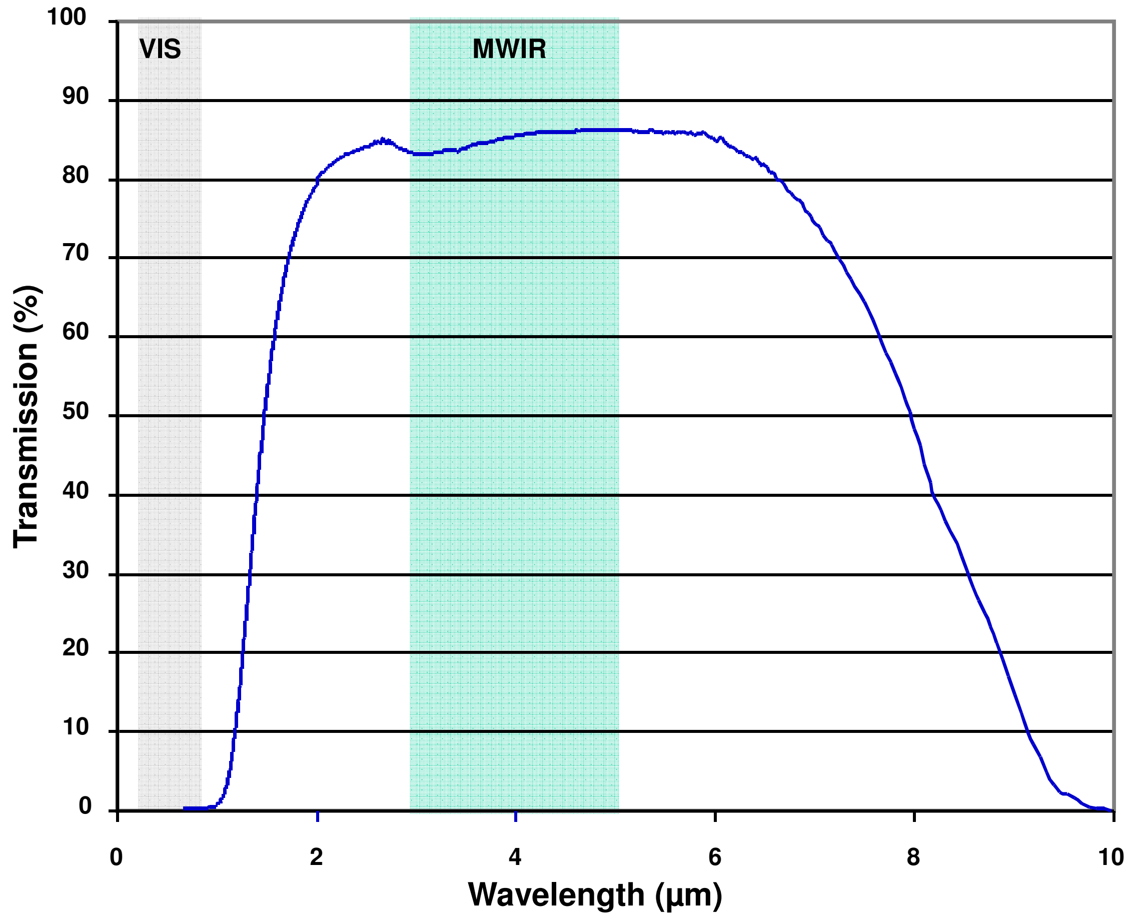
$MgO:Y_2O_3$ Nanocomposites

In the visible band



In the MWIR band





Powder Process Optimization

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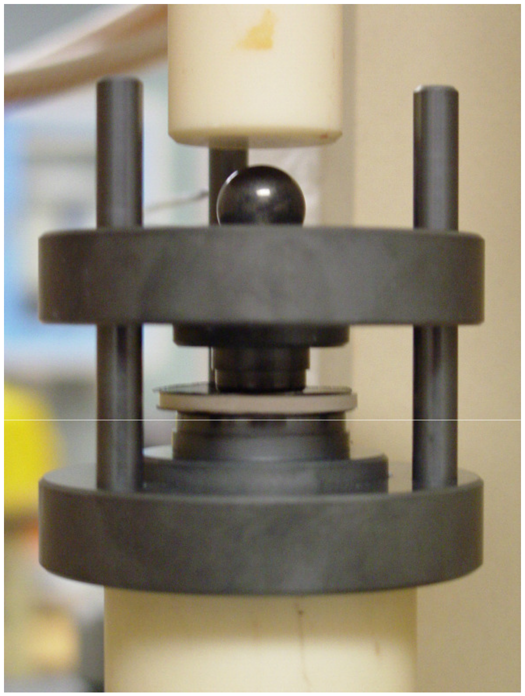

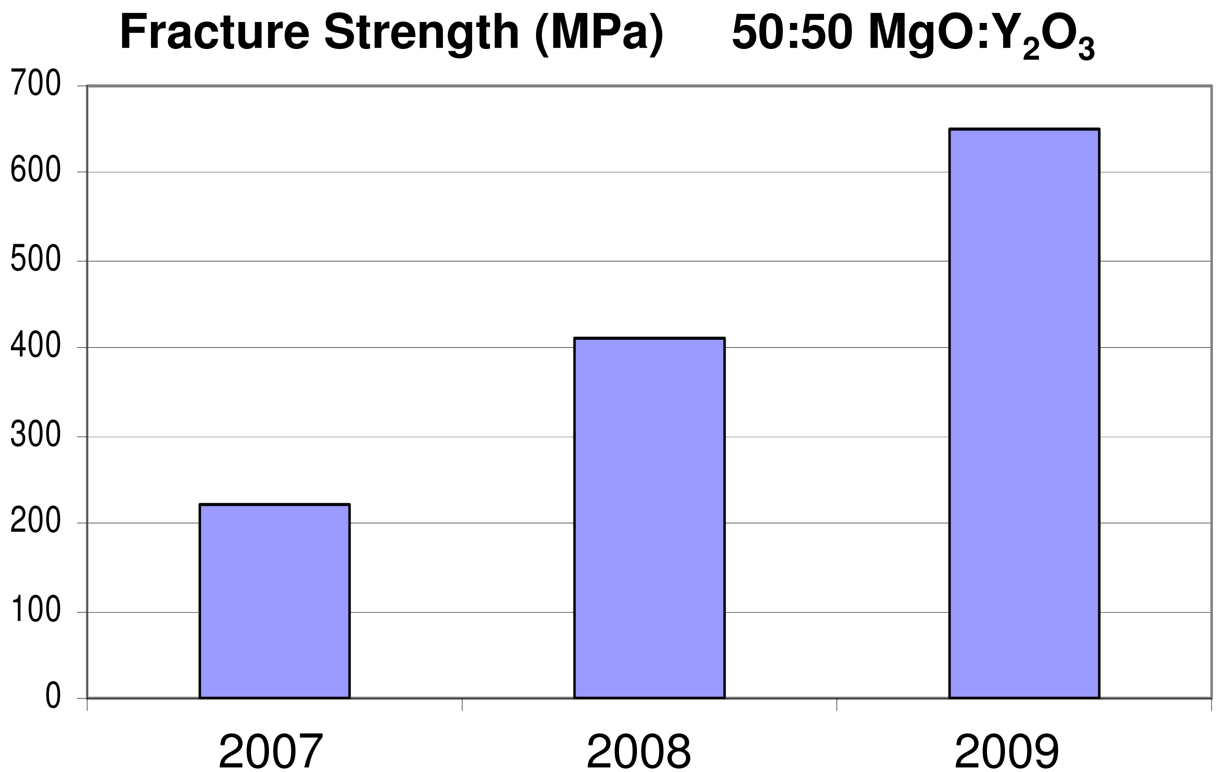

- **Fracture strength improved with optimized powders and processing.**
- **New material systems and/or more energetic processing needed for 1200 MPa!**

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Material Property Goals

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Material Property Metrics for 3-5 micron Nano-Composite Optical Ceramics

Material Property	Units	Phase I Metrics	Achieved	Phase II Metrics
Absorption Coefficient (ave, 3-5µm)	cm^{-1}	≤ 0.1	0.05	≤ 0.1
Optical Scatter (Fwd TIS @ 3.39µm)	%	≤ 2.0	0.6	≤ 0.5
Fracture Strength at 600°C (average of 10 biaxial disks)	MPa	≥ 600	650	≥ 1200
Hardness (µ-indent: 50g load)	kg/mm^2	≥ 2200	2350	≥ 2200
Thermal Shock Resistance (requires thermal conductivity measurement)	calculated FoM: R'	- - -	1.3 X Sapphire	≥ 2X Sapphire
Sand Erosion Resistance (blowing sand – conditions TBD)	grams/std test	- - -		≥ 2X Sapphire
Water Drop Threshold Velocity (Marshall SFC – 3mm drop)	m/s	- - -		≥ 2X Sapphire

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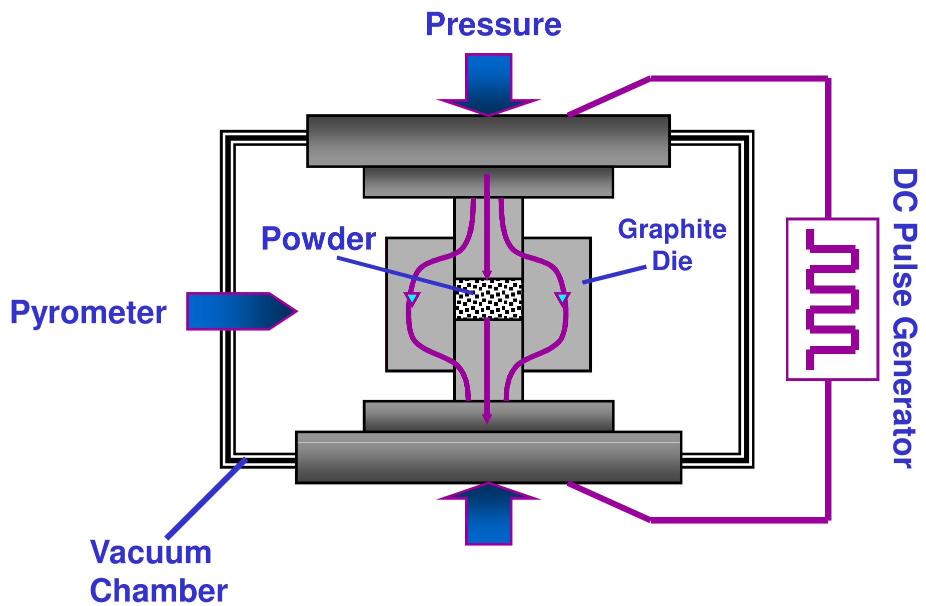
Spark Plasma Sintering



Pulsing Electric Field → **Joule Heating** → **Low T, Fast Sintering**

Spark Plasma Sintering

UC DAVIS Chemical Engineering & Materials Science

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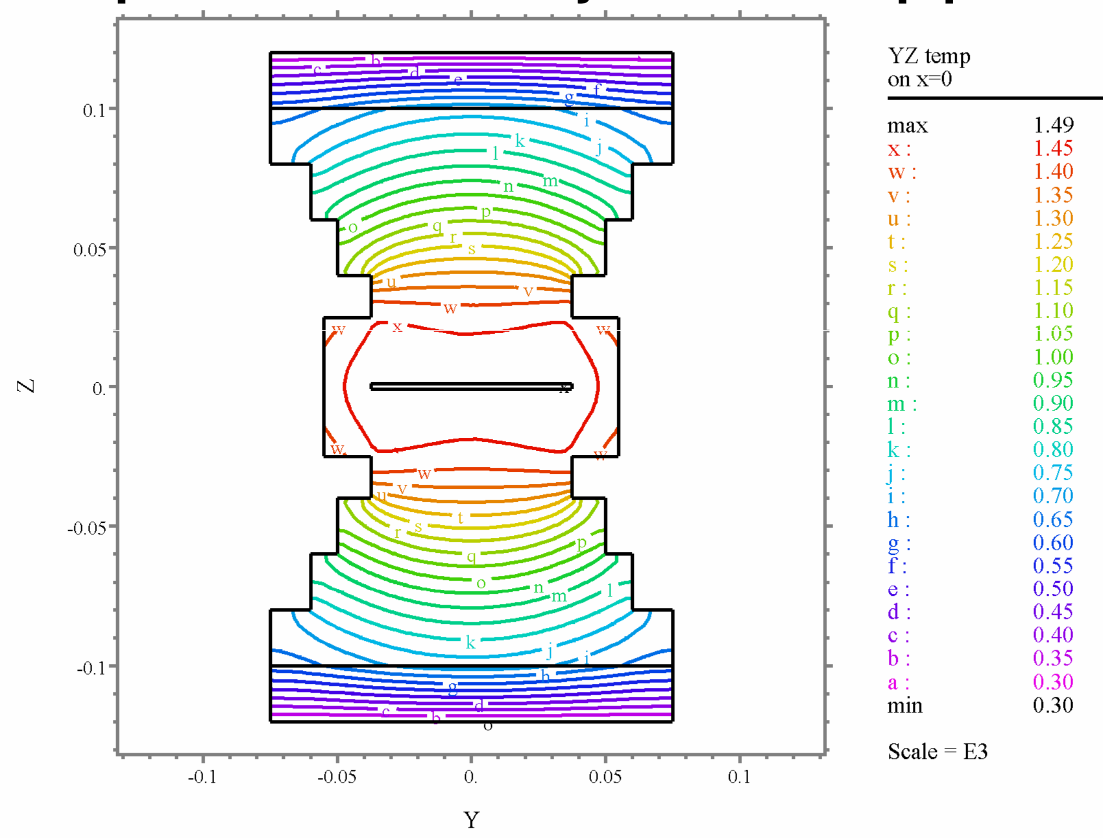
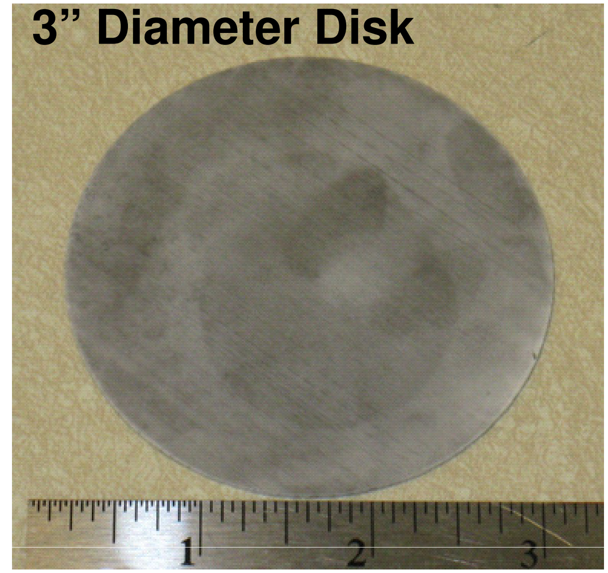
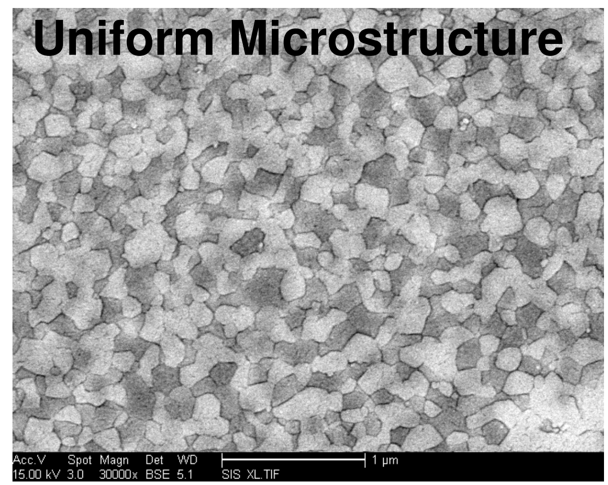
Modeling die geometries to improve temperature uniformity in scaled-up process







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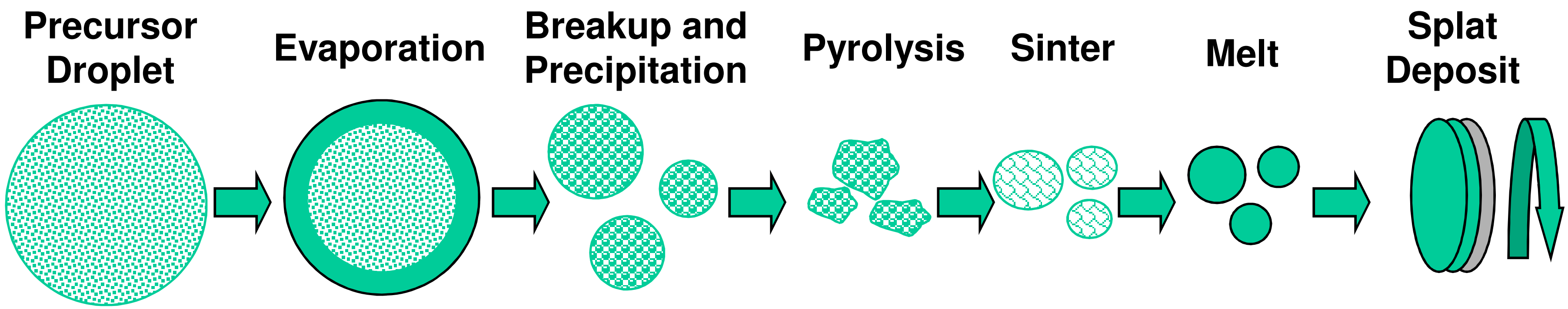
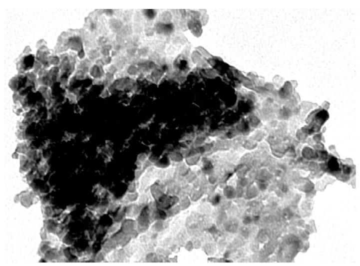
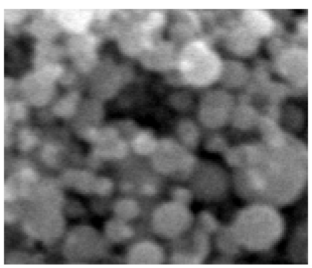
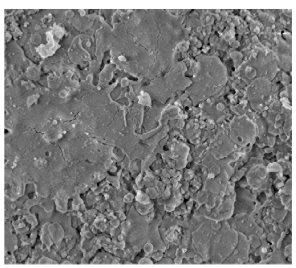
Direct Deposition



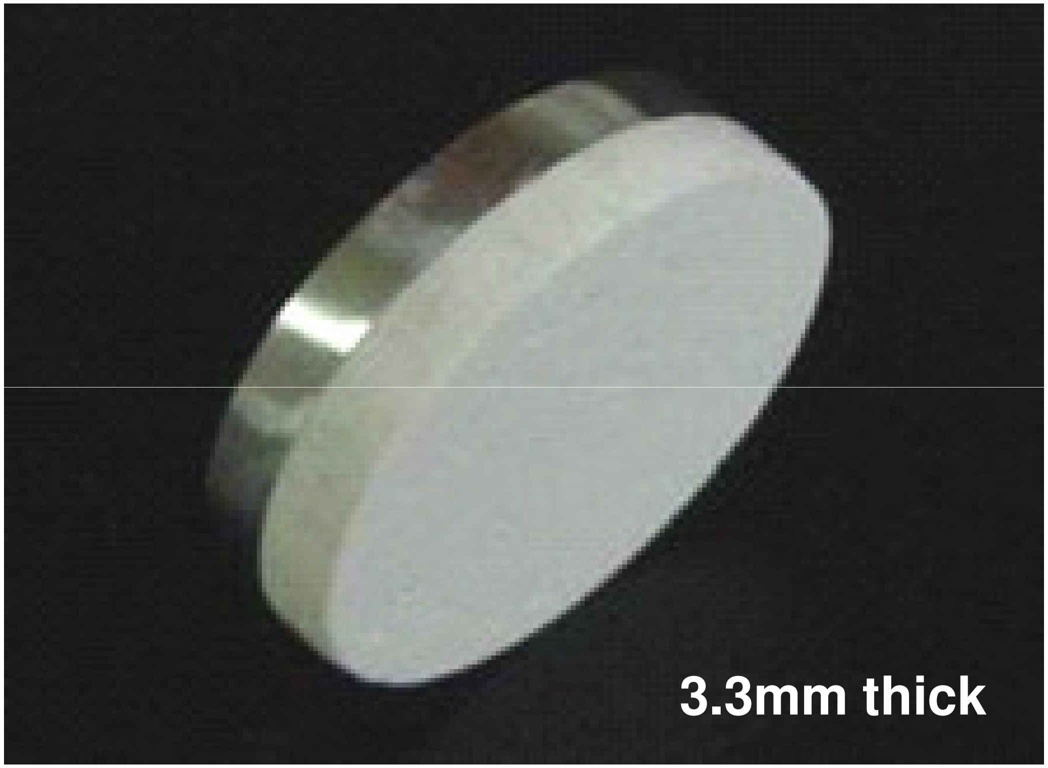


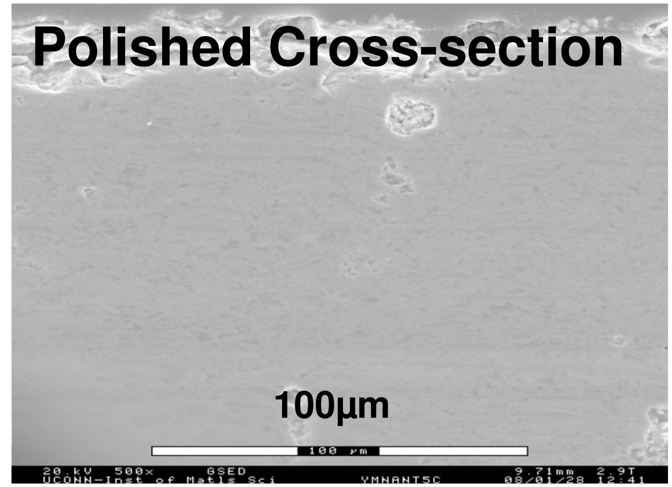


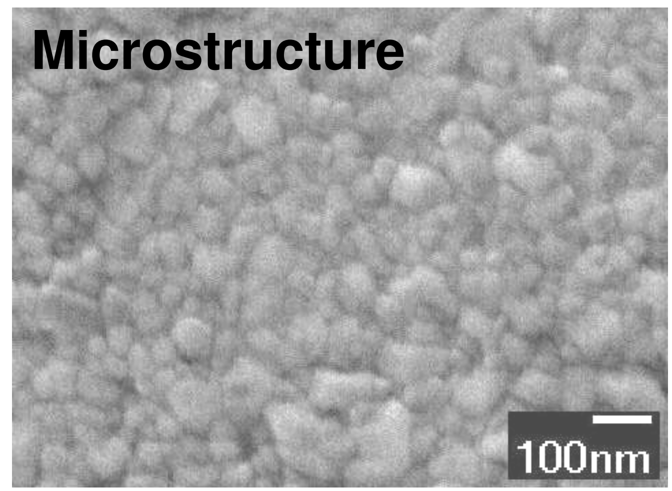


Direct Deposition

50:50 $MgO:Y_2O_3$







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Summary

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- MgO:Y_2O_3 based nanocomposite ceramics have been developed using traditional ceramic processing routes and demonstrated:
  - Sapphire equivalent mechanical durability
  - Yttria equivalent MWIR optical transparency

- New nanocomposite material systems show potential for greater mechanical durability with inherently more durable crystallographic phases.

- More energetic fabrication techniques are showing promise for refined microstructures and improved mechanical properties.